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Description

U.S. Patent 2,656,508 issued 20th October, 1953 describes a system, known as the resistive pulse technique, for counting particles suspended in an electrically conducting fluid medium. An electrically insulating wall, including an aperture several to several hundred micrometers in diameter, is immersed in the fluid. A pair of electrodes are disposed in the fluid on opposite sides of the wall to establish a current path between them through the fluid and passing through the aperture. The fluid is caused to pass through the aperture at a known rate, and an electric current is passed between the two electrodes. The aperture and the resulting constricted electric field in and around it constitute a scanning zone.

While pure fluid is passing through the aperture, the resistance between the two electrodes, and in consequence also the voltage, is steady. When a particle suspended in the fluid passes through the aperture (or, more accurately, through the scanning zone), the resistance is altered, being increased if the electrical conductivity of the particle is lower than that of the fluid as is generally the case. Under constant current conditions, the passage of a suspended particle through the aperture gives rise to a voltage pulse which is readily detected. Alternatively, given constant voltage conditions, the passage of a suspended particle gives rise to a current pulse, in this case a transient reduction in current. Knowing the rate of fluid flow and the rate of occurrence of voltage pulses it is easily possible to calculate the number of particles per unit volume of fluid. Since 1953, the system has been greatly developed and refined and a large volume of patents and literature now exists in respect of it.

Various patents including U.S. 3,668,531 describe how it is possible to use the amplitude of each voltage pulse to determine the size of the particle responsible. U.S. Patent 3628140 provides a conical chamber upstream and/or downstream ofthe aperture in order to increase resolution.

U.S. Patent 3,441,848 is concerned with measuring the lengths of fibres. Fibres suspended in a fluid pass through an aperture lengthwise, and the patent takes advantage of this to use the duration of each voltage pulse to determine the length of the fibre responsible. In U.S. 3,890,568, a combination of voltage pulse amplitude and duration are used to provide a more accurate measure of fibre length.

The system thus far described has been essentially concerned with aqueous fluids, having ionic conductivity characteristics. By European Patent Specification 119770 the concept has been extended to molten metals, having electronic conductivity characteristics, for which purpose a high current density is required.

In all these systems, the cross-sectional area of the aperture has been constant along its length, or has been shaped at its upstream and downstream ends to achieve non-turbulent flow of fluid, but has not been caused to change along its length in any systematic way in order to provide additional information.

According to the present invention, the aperture has a finite length in the direction of flow of the fluid and the cross-section of the aperture is caused to change progressively along at least part of its length. As a result of this progressive change in cross-section, the duration of a resistive pulse caused by passage of a particle through the aperture depends on the size of the particle. According to the characterising parts of claims 1 and 3, the duration of resistive pulses is analysed and provides information about the particle size. By resistive pulse is meant either a transient increase in voltage at constant current or a transient decrease in current at constant voltage. In the preferred mode of practising the invention, constant current conditions are used, and the length of time the voltage exceeds an arbitrary threshold value is measured.

The progressive change in cross-section can be achieved by providing a truncated cone-shaped aperture. Alternatively the aperture can be shaped like the hole in a torus. Other shapes are possible.

Figure 1 is a schematic diagram of apparatus according to the invention,

Figure 2 represents a cone, and

Figures 3 to 6 are axial sections through alternative apertures for use in the equipment of Figure 1.

Figure 7 is a graph of signal against time showing the profile of voltate pulses at different particle sizes.

Referring to Figure 1, there is provided an electrically insulating wall 10 including an aperture 12 therethrough, a pair of electrodes 14, 16 disposed on opposite sides of the wall, all immersed in an electrically conducting fluid having surfaces 18, 20 on opposite sides of the wall. The fluid is caused to pass through the aperture in the direction of the arrow 22 at a controlled flow rate. A constant current is passed between the electrodes, and voltage pulses caused by particles passing through the aperture are detected and analysed (by means not shown).

The aperture is shown as having the shape of a truncated cone having a longitudinal axis 24 and a diameter that decreases in the direction of flow of the fluid.

It can be shown (Deblois R.W. and Bean C.P. "Counting and Sizing of Sub-Micron Particles by the Resistive Pulse Technique." Rev. Sci Instr. Vol. 41 No. 7 Pp 909-915, 1970) that, when a substantially non-conducting particle passes through an opening of circular cross-sectional area (substantially larger than the

cross-section of the particle), the resistance measured across the opening will be given by the following equation:

$$\Delta R = \left(\frac{\Delta V}{I}\right) = \frac{4\rho}{\pi} \cdot \frac{d^3}{D^4} \qquad (1)$$

Where

- ΔR: is the change in the electrical resistance of the opening
- ΔV: is the corresponding change in voltage manifested in the presence of a current I
- ρ: is the resistivity of the fluid
- d: is the equivalent spherical diameter of the particle and
- D: is the diameter of the opening.

By re-arranging equation 1 one obtains

$$D = \left[\frac{4\rho}{\pi}\left(\frac{I}{\Delta V}\right) d^3\right]^{1/4} = \left[\frac{4\rho}{\pi} \cdot \frac{d^3}{\Delta R}\right]^{1/4} \qquad (2)$$

which is an explicit expression for the opening diameter "D" at which a particle of diameter "d" will cause a change in the resistance of the opening of magnitude

$$\Delta R, \left(\frac{\Delta V}{I}\right)$$

By examining Figure 1 and equation (2), it is readily apparent that a larger particle will cause the resistance to change by an amount of ΔR earlier in its passage through the opening than will a smaller particle. Furthermore, as will be shown next, if the geometry of the opening is known and the flow conditions are appropriately chosen it is possible to establish a functional relationship between the duration over which the passage of a particle causes a minimum change in resistance and the dimension of the particle.

The case of an opening having the shape of a truncated cone serves as a convenient example.

Consider the case of a particle of diameter "d" which gives rise to a change in resistance ($\Delta V/I$) at some point "X" within the shaded portion of the cone shown in Figure 2.

Fixing the origin at the apex of the cone, the base diameter "D" is simply:

$D(X) = KX \qquad (3)$

where K is twice the tangent of the $\frac{1}{2}$ angle (θ) subtended by the cone

i.e. $D(X) = 2(\tan\theta)\,X \qquad (4)$

The volume of a cone is known to be:

$$Vol = \frac{1}{3}\pi\left(\frac{D}{2}\right)^2 h \qquad (5)$$

where

h : the altitude of the cone

D : base diameter

Thus the volume of the truncated cone shown in Figure 2 extending from X_1 to some distance X is:

$$V(X) = \frac{\pi}{3} \cdot X\left(\frac{D(X)}{2}\right)^2 - \frac{\pi}{3} \cdot X_1\left(\frac{D(X_1)}{2}\right)^2 \qquad (6)$$

substituting for D(X) = tan θ(X) gives

$$V(X) = \frac{\pi}{3} \cdot \tan^2\theta \cdot (X^3 - X_1^3) \qquad (7)$$

One may assume that there are no substantial velocity gradients within the fluid passing through the truncated cone. This is justified based on the observation that the volumetric flow rate through a small, thin opening can be predicted accurately by Bernoulli's Equation when the assumption of inviscid flow is made. With this assumption, the time "T" that it takes for the particle to pass from point X to point X_1 will be simply

$$T = \frac{V(X)}{Q} \qquad (8)$$

where Q is the volumetric flow rate of the liquid

Substituting equation (2) into equation (4) into equation (8) provides an explicit equation giving the time over which a particle of diameter "d" will cause an increase in the electrical resistance ΔR of the opening of the particle diameter "d":

$$T = \frac{\pi}{3} \cdot \frac{\tan^2\theta}{Q} \cdot \left(\left[\frac{1}{2\tan\theta}\left(\frac{4\rho}{\pi} \cdot \frac{d^3}{\Delta R}\right)^{1/4}\right]^3 - X_1^3\right) \qquad (9)$$

or, more conveniently as in the preferred embodiment, in terms of the voltage change (ΔV) in the presence of an applied current I:

$$T = \frac{\pi}{24 \cdot \tan\theta \cdot Q}\left[\left(\frac{4\rho \cdot I \cdot d^3}{\pi \cdot \Delta V}\right)^{3/4} - Di^3\right] \qquad (10)$$

where Di is the diameter of the smaller end of the truncated cone.

As an example consider the case of molten aluminium (ρ = 25 x 10^{-8} Ωm) being drawn through a truncated conical opening of inferior diameter Di = 3 x 10^{-4} m and taper angle Θ = 8.53 degrees, at the rate of 2.67 x10^{-7} m^3/sec (16 cm^3/min) in the presence of an applied current (I) of 60 amperes. The time T during which the threshold voltage (ΔV, in this case 10 μV) is exceeded during the passage of a particle of

diameter (d) can be obtained by substitution into equation 10:

$d(10^{-6}m)$	$T(10^{-6}s)$
20	54
25	146
30	265
35	412
40	589
45	792
50	1030

The length of the aperture, in the direction of flow of the fluid, should be substantially greater than the length of individual particles, but sufficiently small that the aperture (or the sensing zone associated with it) generally contains not more than one particle at a time. With acicular particles such as fibres, the above equations apply only with somewhat less accuracy, and the invention is thus particularly useful where the suspended particles, as is generally the case with particles suspended in molten metals, do not differ markedly from spherical. The minimum diameter and angle of taper may be chosen having regard to the expected size range of the suspended particles. The upstream and downstream ends of the aperture may be profiled to promote streamlined fluid flow.

It is known that the amplitude of a voltage pulse generated by a particle passing through an aperture of constant cross-section can be analysed to provide a measure of particle size, and this technique can be used in conjunction with an aperture of the type shown in Figure 3. In this Figure, an aperture has an upstream section 25 in the form of a truncated cone whose diameter decreases in the direction of the fluid flow (from left to right), and a downstream section 26 in the form of a cylinder. A measurement of the duration of a voltage pulse (above a datum level), as a particle passes through section 24 of the aperture, can be combined with a measurement of the amplitude of the voltage pulse, as the particle passes through section 26, to provide a good determination of particle size.

The aperture of Figure 3 could alternatively be used with fluid flow from right to left, albeit with somewhat greater risk of turbulence.

In Figure 4, an aperture comprises an upstream section 28 in the form of a truncated cone of decreasing diameter, and a downstream section 30 in the form of a truncated cone of increasing diameter. This profile has the advantage that the change in duration of the voltage pulse for a given change in particle size is doubled in comparison with the profile of Figure 1.

In Figure 5, an aperture has an upstream section 32 in the form of a truncated cone of decreasing diameter, in intermediate section 34 in the form of a cylinder, and a downstream section 36 in the form of a truncated cone of increasing diameter.

In Figure 6, a wall 10 has an aperture 12 having a longitudinal axis 24. But the walls 38 defining the aperture are not straight in a direction parallel to the axis, but are curved. In fact the aperture has the shape of the hole in a torus and may be notionally generated by rotating a circle having a radius r centered at 40 round the axis 24. Alternatively, for the notional circle could have been substituted an ellipse with its major axis parallel or perpendicular to the axis 24 of the aperture. In fact the curved surfaces 38 could have been shaped in a variety of ways consistent with the requirement that the cross-sectional area of the aperture changes progressively along at least part of its length.

Figure 7 is a graphical presentation of the mathematical treatment of the invention. The graph is in the form of theoretical voltage-time curves for the passage of particles of diameters ranging from 20 to 35 microns through a conical aperture having a maximum diameter of 0.6 mm, a minimum diameter of 0.3 mm and a length of 1.3 mm. The curves have been generated by a computer programme using increments of distance, i.e.

V has been calculated using equation (1) under constant current conditions, at increments of 5 micrometers measured along the axis of the aperture. A dashed line has been drawn to illustrate the fact that the duration for which the voltage pulse exceeds a given threshold value, say 10 microvolts, increases

with increasing particle size.

Example

An experiment was performed using equipment including a conical aperture as in Figure 1 with dispersion of glass beads of known diameter in 1% w/w KCl solution in water. The following table sets out the durations of voltage pulses (in milliseconds above an arbitrary reference voltage) against the diameters of the beads.

Voltage Pulse Duration (ms)	Bead Diameter micrometers
0.18	57
0.48	85
0.60	107
0.64	103
0.59	103
0.18	56.8
0.20	56.8

The first three results were obtained using mixed beads in a single dispersion. It is apparent that the voltage pulse durations can be used to determine the bead diameters.

Claims

1. Apparatus for studying particles suspended in an electrically conducting fluid comprising an electrically insulating wall (10) including an aperture (12) therethrough, a pair of electrodes (14,16) disposed on opposite sides of the wall (10) to establish a current path between them through the fluid and passing through the aperture (12), means for causing the fluid to pass at a controlled rate through the aperture (12) and for simultaneously passing an electric current between the two electrodes (14,16) by means of the said current path, and means for detecting resistive pulses representative of changes in electrical resistance across the wall (10) resulting from particles suspended in the fluid passing through the aperture (12), the aperture (12) having a finite length in the direction of flow of the fluid (22) and the cross-sectional area of the aperture (12) changing progressively along at least part of its said length, characterised in that detecting means includes means for analysing the duration of the resistive pulses to provide information about the particle size.

2. Apparatus as claimed in claim 1, adapted for use under constant current conditions, wherein the means for detecting changes in electrical resistance are means for detecting transient voltage increases.

3. A method of studying particles suspended in an electrically conducting fluid comprising establishing a sensing region within the fluid, which sensing region has a longitudinal axis and a cross-sectional area, causing an electric current to flow along a current path extending longitudinally of the sensing region, causing the fluid to flow at a controlled rate in a longitudinal direction through the sensing region, and detecting resistive pulses in the electrical resistance of the fluid passing through the sensing region resulting from the presence therein of said particles, the sensing region having a finite length in a

longitudinal direction, the cross-sectional area of the sensing region changing progressively along at least part of the said length, characterised in that detecting resistive voltage pulses includes analysing the duration of the said pulses to provide information about the particle size.

4. A method as claimed in claim 3, wherein the electrically conducting fluid is a molten metal.

5. A method as claimed in claim 3, wherein current is caused to flow under constant current conditions, and changes in electrical resistance are detected in the form of transient voltage increases.

Revendications

1. Appareil pour l'étude de particules en suspension dans un fluide électriquement conducteur, cet appareil comprenant une paroi électriquement isolante (10) qui comporte une ouverture (12) qui la traverse, une paire d'électrodes (14, 16) qui sont placées sur des côtés opposés de la paroi (10) afin d'établir une voie de courant entre elles au travers du fluide, cette voie de courant traversant l'ouverture (12), un moyen pour forcer le fluide à traverser l'ouverture (12) selon un débit contrôlé et pour simultanément faire passer un courant électrique entre les deux électrodes (14, 16) au moyen de ladite voie de courant et un moyen pour détecter des impulsions résistives qui sont représentatives de variations de la résistance électrique au travers de la paroi (10), ces variations résultant des particules qui sont en suspension dans le fluide et qui traversent l'ouverture (12), l'ouverture (12) ayant une longueur finie suivant la direction de l'écoulement du fluide (22) et la section en coupe transversale de l'ouverture (12) changeant progressivement le long d'au moins une partie de ladite longueur de l'ouverture, cet appareil étant caractérisé en ce que le moyen de détection comporte un moyen pour analyser la durée des impulsions résistives afin de fournir une information relative à la taille des particules.

2. Appareil selon la revendication 1, cet appareil étant étudié pour fonctionner à des conditions de courant constant, dans lequel le moyen qui permet de détecter des variations de la résistance électrique est un moyen qui permet de détecter des accroissements de tension transitoires.

3. Procédé pour l'étude de particules en suspension dans une fluide électriquement conducteur, ce procédé comprenant l'établissement d'une région de détection à l'intérieur du fluide, laquelle région de détection a un axe longitudinal et une section en coupe transversale, ce procédé consistant à forcer l'écoulement d'un courant électrique le long d'une voie de courant qui s'étend longitudinalement par rapport à la région de détection, à forçer le fluide à s'écouler selon un débit contrôlé suivant une direction longitudinale au travers de la région de détection et à détecter des impulsions résistives dans la résistance électrique du fluide qui traverse la région de détection, ces impulsions résistives résultant de la présence dans le fluide desdites particules, la région de détection ayant une longueur finie suivant une direction longitudinale, la section en coupe transversale de la région de détection variant progressivement le long d'au moins une partie de la longueur de ladite région, ce procédé étant caractérisé en ce que la détection des impulsions de tension résistives inclut l'analyse de la durée desdites impulsions pour fournir une information relative à la taille des particules.

4. Procédé selon la revendication 3 dans lequel le fluide électriquement conducteur est un métal en fusion.

5. Procédé selon la revendication 3 dans lequel le courant est forcé à s'écouler à des conditions de courant constant et dans lequel des modifications de la résistance électrique sont détectées sous la forme d'augmentations de tension transitoires.

Patentansprüche

1. Vorrichtung zum Nachweisen von in einem elektrisch leitenden Fluid gelösten Teilchen, umfassend: eine elektrisch isolierende Wand (10), die eine Öffnung (12) in dieser aufweist, ein Elektrodenpaar (14, 16), das auf gegenüberliegenden Seiten der Wand (10) angeordnet ist, um einen Strompfad zwischen diesen durch das Fluid und durch die Öffnung (12) hindurch herzustellen, eine Einrichtungen, die veranlaßt, daß das Fluid mit einer gesteuerten Rate durch die Öffnung (12) hindurchtritt, und die gleichzeitig einen elektrischen Stromzwischen den beiden Elektroden (14, 16) über den Strompfad

fließen läßt, und Einrichtungen zum Ermitteln von Widerstands-Impulsen, welche Veränderungen im elektrischen Widerstand über die Wand (10) aufgrund von in dem Fluid gelösten Teilchen darstellen, welche durch die Öffnung (12) hindurchtreten, wobei die Öffnung (12) eine endliche Länge in der Strömungsrichtung des Fluides (22) hat, und die Querschnittsfläche der Öffnung (12) sich zunehmend im Verlauf wenigstens eines Teiles ihrer Länge verändert,
dadurch **gekennzeichnet,** daß die Ermittlungseinrichtung Einrichtungen zum Analysieren der Dauer der Widerstandsimpulse enthält, um Information über die Teilchengröße zu liefern.

2. Vorrichtung nach Anspruch 1, ausgelegt zur Verwendung unter Konstantstrombedingungen,
dadurch **gekennzeichnet,** daß die Einrichtungen zum Ermitteln von Veränderungen im elektrischen Widerstand Einrichtungen zum Ermitteln von transienten Spannungszuwächsen sind.

3. Verfahren zum Nachweisen von in einem elektrisch leitenden Fluid gelösten Teilchen, welches umfaßt: Aufbau einer Abfühlzone in dem Fluid, welche Abfühlzone eine Längsachse und eine Querschnittsfläche hat, Fließenlassen eines elektrischen Stromes über einen sich longitudinal durch die Abfühlzone erstreckenden Strompfad, Fließenlassen des Fluides mit einer gesteuerten Strömungsrate in einer Längsrichtung durch die Abfühlzone, und Ermitteln von Widerstandsimpulsen im elektrischen Widerstand des die Abfühlzone passierenden Fluides, welche vom Vorhandensein der Teilchen in diesem resultieren, wobei die Abfühlzone eine endliche Länge in einer Längsrichtung hat, und die Querschnittsfläche der Abfühlzone sich zunehmend entlang wenigstens eines Teiles der Länge verändert,
dadurch **gekennzeichnet,** daß das Ermitteln von Widerstandsspannungsimpulsen das Analysieren der Dauer der Impulse einschließt, um Informationen über die Teilchengröße zu liefern.

4. Verfahren nach Anspruch 3,
dadurch **gekennzeichnet,** daß das elektrisch leitende Fluid ein geschmolzenes Metall ist.

5. Verfahren nach Anspruch 3,
dadurch **gekennzeichnet,** daß veranlaßt wird, daß Strom unter Konstantstrombedingungen fließt und daß Veränderungen im elektrischen Widerstand in der Form von transienten Spannungszuwächsen ermittelt werden.

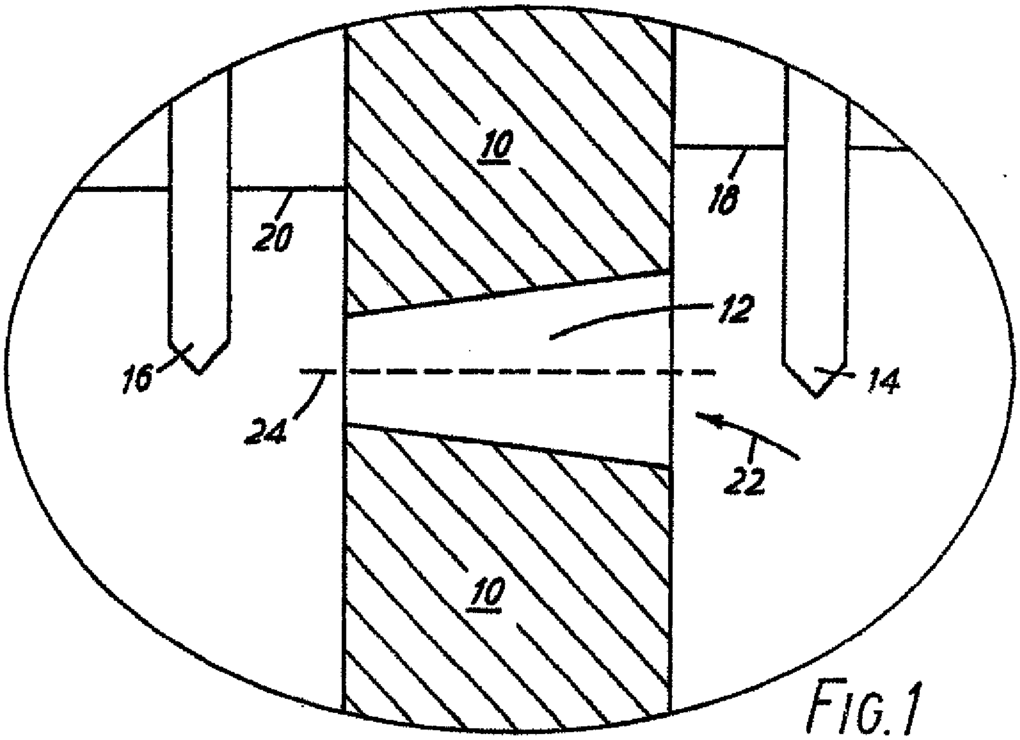

FIG.1

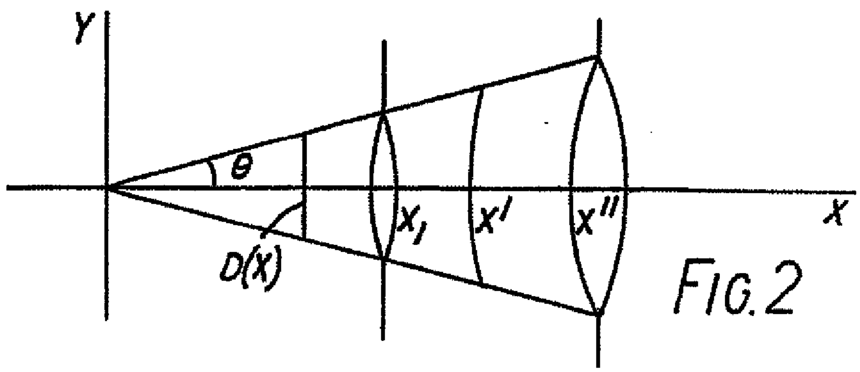

FIG.2

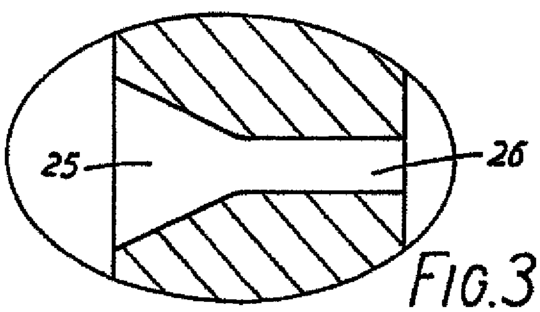

FIG.3

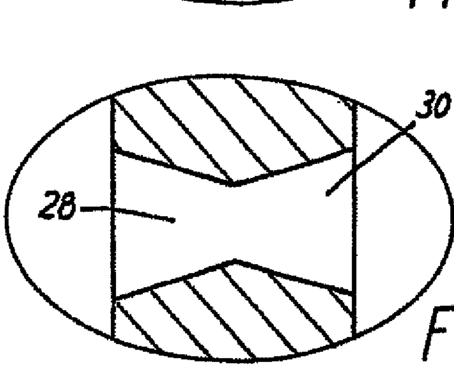

FIG.4

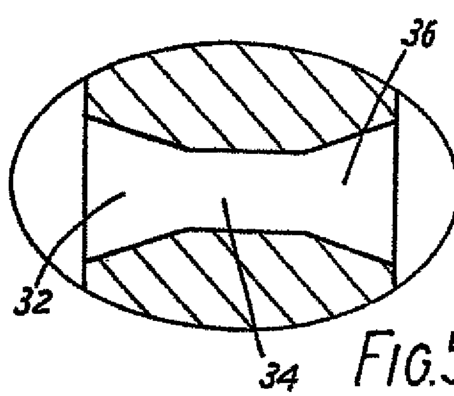

FIG.5

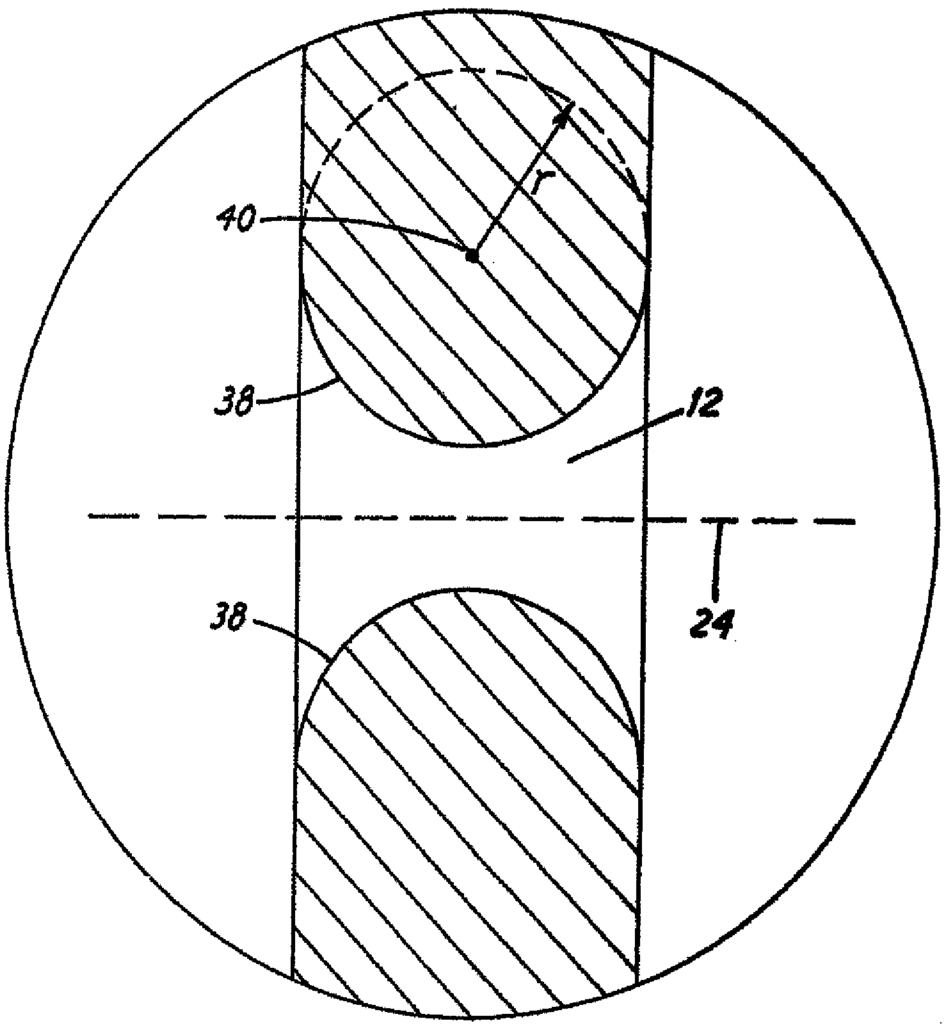

FIG. 6

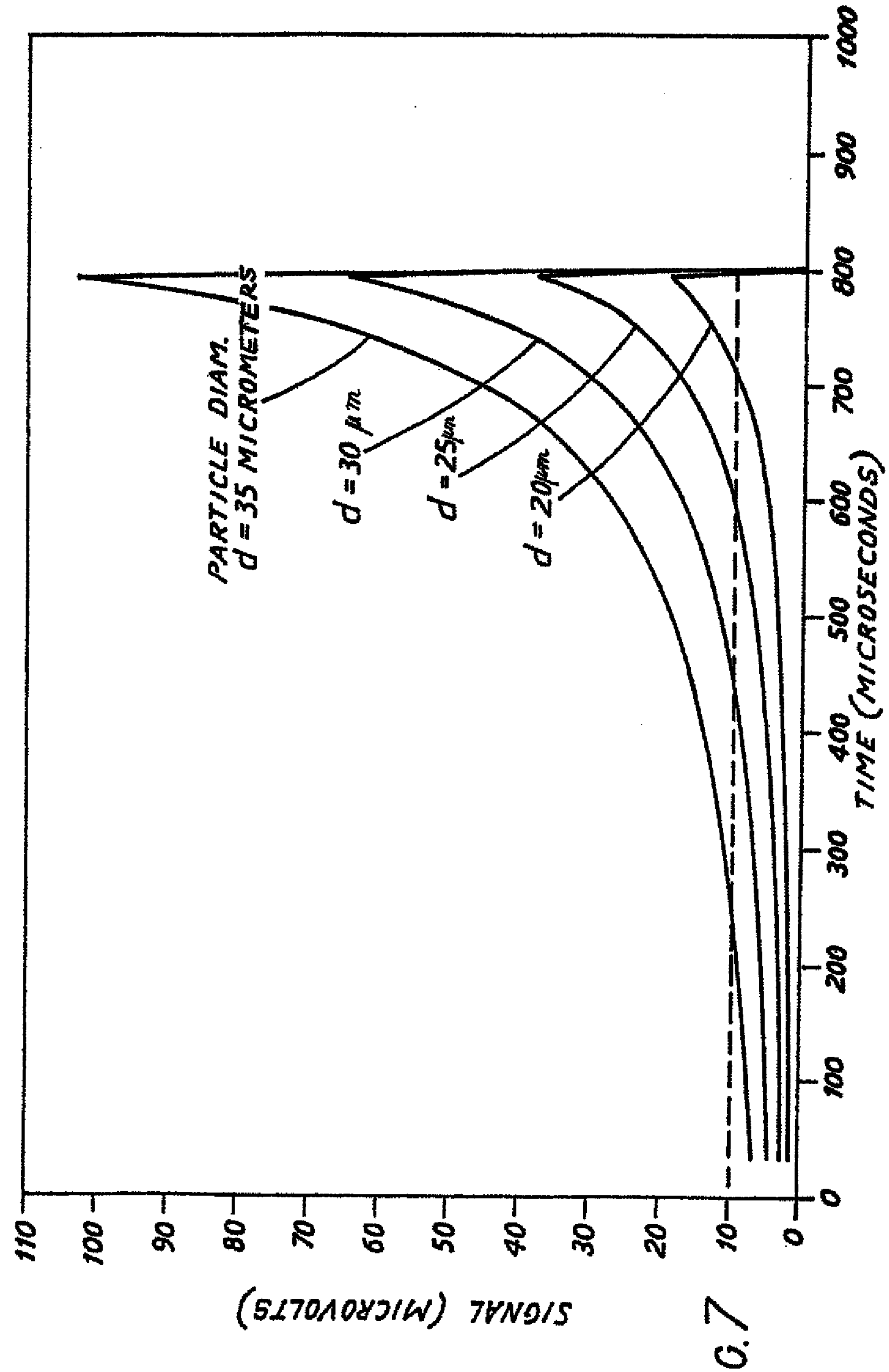
PARTICLE DIAM.
d = 35 MICROMETERS
d = 30 μm
d = 25μm
d = 20μm
TIME (MICROSECONDS)
SIGNAL (MICROVOLTS)
0
100
200
300
400
500
600
700
800
900
1000
10
20
30
40
50
60
70
80
90
100
110

FIG. 7